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UNIVERSITY OF LOUISVILLE

THE EFFECT OF ETHYL CELLULOSE
ON THE PROPERTIES OF
SHORT-OIL VARNISHES

A Thesis

Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

John Bradford Scott

1940

THE EFFECT OF ETHYL CELLULOSE
ON THE PROPERTIES OF
SHORT-OIL VARNISHES

John Bradford Scott

Approved by Examining Committee:

Director

May 25, 1940

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DEFINITIONS AND SYMBOLS

Varnish Length. The number of gallons of oil used for each one hundred pounds of resin in a varnish is designated the length of the varnish.

Set-to-touch Time. The set-to-touch time of a varnish is defined as the time required after application to a glass panel for the varnish to be sufficiently dry so that no varnish will adhere to the finger when the surface of the varnish is rubbed lightly.

Tack-free Time. The tack-free time of a varnish is the time required after application of the varnish for the disappearance of all tackiness or stickiness when the finger is pressed firmly on the surface of the varnish.

The composition of each varnish used in this investigation will be indicated by its code symbol. The letter V, indicating varnish, will be followed by two series of numbers separated by dashes. The first series of numbers will indicate the phenolic resin content expressed as a percentage of the total resin present in the varnish. It may be seen by inspection that the ester gum content is determined by the difference between 100 and the phenolic resin content. The second series of

numbers will indicate the ethyl cellulose content expressed as a percentage of the total non-volatile matter in the varnish. For example, the code symbol V-15-6 will indicate that the resin is composed of 15% phenolic resin and 85% ester gum, and that 6% of the total non-volatile material is ethyl cellulose.

The following symbols will be used to designate the degree of printing:

N - None	No effect
VS - Very slight	Very shallow and appearing only on small portion of area
S - Slight	Shallow indentations in film
C - Considerable	Medium indentations in film
B - Bad	Deep indentations in film
VB - Very bad	Deep and rough indentations in film

The following symbols will be used to designate the condition of the varnish films after the water and alcohol tests:

H -Hard
VSS - Very slightly soft
SS - Slightly soft
S - Soft
VS - Very soft

The following symbols will be used to designate the adhesion and the flexibility of the varnish after stated drying times and temperatures:

G - Good

F - Fair

P - Poor

VP - Very poor

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ABSTRACT

The effect of the incorporation of various percentages of ethyl cellulose on the principal properties of ten-gallon-long phenolic resin - ester gum - china wood oil varnishes has been investigated. The presence of as little as 4% of ethyl cellulose definitely increases the drying rate of the varnish and improves the adhesion, print, water, and alcohol resistances of the dried film. Increasing amounts of ethyl cellulose, up to 12%, cause proportionate improvements in the same properties. The presence of ethyl cellulose in the film materially increases the flexibility of the aged film.

INTRODUCTION

The purpose of this investigation was to show the effect of the addition of ethyl cellulose to varnishes of short oil-length for primary use in furniture finishes.

Ethyl cellulose is one of the most recent cellulose derivatives of commercial importance. It has already found use in the plastic, wrapping and protective coating industries, and has shown a great deal of promise in many other fields. A very important use in the protective coating industry has been in lacquers. Because of its success in lacquers, much work has been done to adapt ethyl cellulose for use in varnishes.

This investigation was accomplished by making china wood oil varnishes of a definite length containing various proportions of a phenolic resin, ester gum, and ethyl cellulose so that all feasible proportions of these materials were included or could be interpolated from the results. The properties of each varnish were determined and the results tabulated and represented by means of graphs and curves. The properties investigated included set-to-touch time, tack-free drying time, print resistance, water resistance, alcohol resistance, and the effect of aging on flexibility and adhesion.

The results of all dry tests have been plotted

in the form of curves, and the effect of varying the composition of the varnishes on the print resistance has been shown by means of bar graphs. The time required for the attainment of reasonable print resistance of the various varnishes under specific drying conditions has been shown by curves.

HISTORICAL

Probably one of the first descriptions of ethyl cellulose and methods for its preparation appeared in 1905 in the *Monatshefte fuer Chemie* by Suida (18). Leuchs (14), Lilienfeld (15), Dreyfus (5) and many others obtained patents on the preparation of the ether. Denham (3) and his co-workers also had shown that the cellulose ethers may be formed by the substitution of an alkyl or aryl radical in place of a hydroxyl hydrogen atom in cellulose. The high cost of manufacturing the ethers at that time overcame the many industrial possibilities that were indicated.

Until 1936, all ethyl cellulose used in the United States was imported, but commercial ethyl cellulose made by American manufacturers at a lower cost than the imported material is now upon the market.

The manufacture of ethyl cellulose (11, 13) follows the classical methods for the preparation of cellulose ethers. Cellulose from any desired source, such as cotton or wood, is converted into alkali cellulose by treatment with a strong aqueous solution of sodium hydroxide. This alkali cellulose is alkylated with such reagents as ethyl chloride or sulfate. The temperature at which this alkylation is carried out must be controlled to prevent undesirable degradation and destruction, both of the cellulose and of the alkylating agent. After the ether-

ification is complete the excess reagents and the by-products of the reaction are removed by washing and distillation. The purification of ethyl cellulose is a simple operation and consists essentially of washing the product free from soluble materials.

The properties of ethyl cellulose (11, 13, 21), as those of other cellulose derivatives, depend primarily upon the degree to which the cellulose has been etherified. It is customary to consider the cellulose molecule as composed of glucose residues, each of which has three hydroxyl groups available for etherification. The ethyl cellulose of commerce has somewhat less than three of the available hydroxyl groups etherified.

The effects of variation in the degree of ethylation are very pronounced with respect to the solubility of the product in water and organic solvents, and with respect to the melting point of the ether.

Solubility in both polar and non-polar organic solvents is best with ethyl cellulose with 47-49.5% ethoxy content, corresponding to 2.4 to 2.58 hydroxyl groups etherified per glucose residue. The commercial ethyl cellulose having this degree of substitution combines the widest variety of useful properties.

In all references to ethyl cellulose throughout this discussion it will be assumed that the material is that containing 2.4 to 2.58 ethoxy groups per glucose residue, known as standard type.

Ethyl cellulose is a white solid, resembling cellulose acetate in its resistance to flame and nitrocellulose in its compatibility with other film-forming materials. Ethyl cellulose is more resistant to acids and alkalies and to discoloration by sunlight than the esters of cellulose (21). Ethyl cellulose shows the widest solubility of any commercially available film-forming material (4,9,10,11,21). Films of ethyl cellulose exhibit a greater flexibility and toughness than the other compounds of cellulose and retain their flexibility at temperatures as low as 40° C (4,9,10,11,21).

Even in low percentages, ethyl cellulose adds a remarkable degree of flexibility to hard resins, both natural and synthetic. The compatibility of ethyl cellulose with resins more closely resembles that of nitrocellulose than that of cellulose acetate. Ethyl cellulose differs from nitrocellulose in that it does not show as wide compatibility with alkyd resins, more especially the unmodified and short oil length alkyds (4,9,10,11,21).

The tolerance of ethyl cellulose for many incompatible resins can be greatly improved by incorporating a third ingredient which is a good mutual solvent for both the ethyl cellulose and the incompatible resin (13).

THEORETICAL

A varnish can best be defined by considering the nature and purpose of its various components. Varnishes have volatile and non-volatile constituents: the non-volatile consisting of drying oils, resins, and, usually, minute amounts of metallic driers, - the volatile consisting of suitable solvents.

The non-volatile portion of a varnish is the film-forming constituent, while the solvent is present to facilitate the application of the non-volatile material in a smooth, unbroken film of uniform thickness.

Hardening and film formation in varnishes are due principally to simultaneous oxidation and polymerization, both these processes taking place at the chemically reactive double bonds. In these cases the hardness and rigidity of the varnish film appear to be roughly proportional to the number of double bonds capable of entering into the polymerization reaction (20).

Some varnishes, especially those containing reasonably large amounts of phenolic resin, fail on adhesion because the tensile strength of the film far exceeds the adhesive strength even though the adhesive strength equals or exceeds that required for perfect adhesion of a more extensible varnish under the same dimensional changes of base or film. Experience has shown that a straight china

wood short oil varnish occasionally manifests too great a rigidity and mechanical strength for the best adhesion (19).

Ethyl cellulose is a film-forming material which forms a film by the evaporation of solvents rather than through polymerization and oxidation as is the case with drying oils. When properly incorporated in a varnish, the molecules of ethyl cellulose do not exert any influence upon the rate of oxidation of the oil (9), but are considered to form a network which acts as a support for the wet film and as a binder for the dry film (17). Such a composition should provide quicker set-up and faster drying of a varnish film, and should increase the toughness and adhesion of the film after drying. This has been found to be true if the ethyl cellulose is not degraded excessively by the method of incorporation (17).

When ethyl cellulose is properly incorporated in the varnish, a colloidal dispersion results which as a viscosity much greater than the varnish alone under the same conditions. Thus it is possible to produce a varnish of high viscosity without cooking the oil to the extent that cross-linkage of the oil-molecules occurs.

Some of the usual varnish solids are excellent solvents for ethyl cellulose, others are somewhat less active, while still others have very slight or no solvent power (9,10,12). To be of any value in a varnish, the ethyl

cellulose must be completely soluble in the varnish solids; if there is incompatibility, there will be no benefit from the use of ethyl cellulose. It will be obvious that the oil and the resin used must be selected carefully to give complete solubility of the ethyl cellulose. The more active solvents (resin or oil) will tolerate greater dilution with non-solvent ingredients than less active solvents.

Heat-bodied oils are generally incompatible with ethyl cellulose, but a number of varnish resins are compatible with ethyl cellulose. Phenolic resins, among others, are active solvents for ethyl cellulose (12).

The solid portion of a conventional cooked varnish is composed of an oil and resin gel, the oil portion of which is heat-bodied and therefore incompatible with ethyl cellulose. The resin portion, however, can be selected to be an active solvent for ethyl cellulose. Sufficient active ethyl cellulose solvent must be used to overcome the incompatibility of the ethyl cellulose with the heat bodied drying oil.

It is obvious, therefore, that short-oil varnishes are more compatible with ethyl cellulose than long-oil varnishes because short-oil varnishes contain less heat bodied oil. Likewise, there is a greater range of resins from which to choose because less active solvent types serve

satisfactorily as mutually compatible blending agents between the oil and the ethyl cellulose.

The increase in film hardness during the first few hours of drying is more pronounced in the shorter oil varnishes. This condition may be explained by the assumption that in the shorter oil lengths a fair proportion of the oil is hardened by the resin present, leaving the ethyl cellulose a smaller ratio to support. In the longer oil varnishes, while the ratio of oil to ethyl cellulose is the same, the degree of hardness supplied by the resin is much less, and the duty of supporting or hardening the film is largely taken over by the ethyl cellulose (17).

In the past many formulators have tried adding ethyl cellulose to their regular cooked varnishes. This has not been a successful procedure because (1) neither resin nor oil was selected for ethyl cellulose compatibility, (2) usually too much heat-bodying of the oil had taken place in cooking, (3) the straight chain hydrocarbon thinners commonly used in regular cooked varnishes are non-solvents for ethyl cellulose; therefore, to use ethyl cellulose in a varnish, the formula must be designed with these three conditions in mind.

The most satisfactory method of rendering ethyl cellulose compatible with varnishes consists of adding the

ethyl cellulose to the varnish on the down heat, and at such a temperature that the batch is cooled to approximately 500° F. If the batch is small, it may be held at this temperature for the clarity of a cold drop. If the batch is large, it will usually cool at a slow enough rate that the ethyl cellulose will be rendered compatible before the temperature of the batch drops to 400° F.

If ethyl cellulose is held in a varnish cook at high temperatures (over 500° F) for more than a few minutes, it will not improve the drying of the resultant varnish (17), probably because the viscosity of the ethyl cellulose is so reduced that the benefit of its film-forming properties is lost. In other words the molecules are shortened to such an extent by the prolonged heating at high temperatures that they cannot form a supporting network.

MATERIALS

The materials used in this investigation were China wood oil, ester gum, phenolic resin, ethyl cellulose, zinc resinate, mineral spirits, aromatic naphtha, lead acetate, and lead and cobalt naphthenates.

The properties of these raw materials are listed below:

China Wood Oil - This oil was imported by the Wah Chang Trading Corp., and had an acid value of 6.0, a specific gravity of 0.940, an iodine number of 162, and a saponification number of 193.

Ester Gum - This semi-synthetic resin was a product of Reichhold Chemicals, Inc., and was termed Synthe-Copal Ester. It had a color of WG-N, an acid value of 6-8, a melting point of 149° - 162° F, and a specific gravity of 1.10 - 1.15.

Phenolic Resin - This was a 100% phenolic resin manufactured by Bakelite Corp. under the name Bakelite BR-3360. It has an acid value of 40, a melting point of 175° - 205° F, and a specific gravity of 1.152 - 1.174.

Ethyl Cellulose - This cellulose ester was supplied by the Dow Chemical Co. under the name of Ethocel. It is of standard ethoxy content (48.5 - 49.5%) and has a viscosity of 10 centipoises. It is a white granular substance having a specific gravity of 1.13.

Zinc Resinate - This material was obtained from General Naval Stores, Inc., under the trade name of Zirex. It has a color of WG, an acid value of 120 - 135, and a melting point of 160° C.

Mineral Spirits - This is a petroleum distillate manufactured by Shell Oil Co. Its specific gravity is 0.788, the distillation range is 310° - 390° F, and the Kauri-Butanol value is 36-39.

Aromatic Naphtha - This solvent was obtained from Shell Oil Co. under the name Shell Solvent TS-11. It has a specific gravity of 0.820, a distillation range of 105° - 170° C, and a Kauri-Butanol value of 73.

Lead Acetate - This was commercial white lead acetate crystals obtained from Harshaw Chemical Co. The lead content was 63%.

Lead and Cobalt Naphthenates - These were solutions of metallic naphthenates as sold by Nuodex Products Co. under the general name of Nuodex Driers. Their metallic contents were, respectively, 24% lead and 6% cobalt.

EXPERIMENTAL PROCEDURE

PREPARATION OF VARNISHES AND PANELS

Three ten-gallon-long china wood oil varnishes were prepared and designated V-5-0, V-15-0, and V-25-0 respectively. A typical formula and cooking procedure was as follows:

<u>V-15-0</u>		
Pounds	Gallons	Material
85		Ester gum
15		Bakelite BR-3360
78.3	10	China wood oil
	16	Mineral spirits
	16	Shell Solvent TS-11
4.42		Nuodex lead 24%
0.333		Nuodex cobalt 6%

Heat the ester gum and 75% of the china wood oil to 400° F. Add the Bakelite and heat to 500° F. Hold at that temperature until the foam subsides then heat to 560° F. Add the balance of the china wood oil, heat to 480° F and hold at that temperature for one hour. Thin with 50% mineral spirits and 50% Shell Solvent TS-11 to a viscosity of D (Gardner-Holdt) or 1.0 poise and add Nuodex driers.

The drier content of these varnishes was 0.01% Cobalt and 0.55% Lead based on the non-volatile portion.

Another series of six varnishes was prepared.

These varnishes were the same as the varnishes described on page 19 except that they contained various amounts of ethyl cellulose expressed as per cents of the total non-volatile material. These varnishes also contained 1% Zirex and 2% lead acetate based on the weight of the oil. The addition of Zirex and lead acetate was made to improve the solubility of the ethyl cellulose in the varnish solids. Because of the addition of the lead acetate the Nuodex Lead was eliminated. These varnishes were:

V-5-6	V-5-12
V-15-6	V-15-12
V-25-6	V-25-12

A typical formula and cooking procedure was as follows:

<u>V-15-6</u>		
Pounds	Gallons	Material
85		Ester gum
15		Bakelite BR-3360
78.3	10	China wood oil
0.783		Zirex
24.5		Ethyl cellulose
1.56		Lead acetate
	27.5	Mineral spirits
	27.5	Shell Solvent TS-11
.338		Nuodex Cobalt 6%

Heat the ester gum, Zirex, and china wood oil to 400° . Add the Bakelite, heat to 500° F and hold at that temperature until the foam subsides. Add the lead acetate, heat to 510° F, and add the ethyl cellulose ten minutes

after adding the lead acetate. Cool to 450° F and hold 15 minutes. Thin with 50% Mineral Spirits and 50% Shell Solvent TS-11 to a viscosity of D (Gardner-Holdt) or 1.0 poise and add Nuodex Cobalt.

The drier content of these varnishes was 0.01% Cobalt and 0.55% Lead based on the non-volatile portion.

Blends of the varnishes containing no ethyl cellulose and 12% ethyl cellulose were made to obtain a series of varnishes containing 4, 6, 8, and 10% of ethyl cellulose.

A typical blend formula was:

V-15-6

Varnish	Pounds
V-15-0	42.55
V-15-12	57.45
	<u>100.00</u>

The non-volatile content of all cooked and blended varnishes at the same viscosity (D - Gardner-Holdt, or 1.0 poise) is shown in Table I.

The panels for the determination of the set-to-touch time and the tack-free time were prepared by flowing the varnishes across 6" x 12" glass panels and standing the

TABLE I

NON-VOLATILE CONTENT OF VARNISHES

<u>VARNISH</u>	<u>% NON-VOLATILE</u>
V-5-0 *	56.4
V-5-4	49.5
V-5-6 (Cook)	53.2
V-5-6 (Blend) #	46.6
V-5-8	44.1
V-5-10	41.8
V-5-12	39.7
V-15-0	50.1
V-15-4	44.8
V-15-6 (Cook)	49.3
V-15-6 (Blend)	42.6
V-15-8	40.6
V-15-10	38.8
V-15-12	37.1
V-25-0	44.0
V-25-4	40.0
V-25-6 (Cook)	46.5
V-25-6 (Blend)	38.3
V-25-8	36.7
V-25-10	35.3
V-25-12	33.9

* V-5-0 is the designation for a varnish (V) with 5% of resin as Bakelite BR 3360 and 95% as ester gum (5) and 0% ethyl cellulose (0).

Blends were prepared from varnishes containing 0% and 12% ethyl cellulose.

panels in a vertical position to drain.

The panels used for the determination of print resistance, water resistance, and alcohol resistance, were of 10" x 16" three-ply walnut veneer. The panels were well sanded, filled with a combination stain and filler and allowed to dry overnight. The panels were then given one coat of a suitable lacquer sealer, sanded after air-drying one hour, and then given a double coat of the varnish to be tested.

One panel of each varnish was allowed to dry at room temperature for twenty-four hours.

One panel of each varnish was baked for one and one-half hours at 150° F and allowed to cool one hour before rubbing.

Seven similar series of panels were force-dried at 120° F for seven different periods of time, from two to eight hours in one hour increments, and allowed to cool one hour before rubbing.

All baking and force-drying was done in an electrically heated industrial drying oven, equipped with an air-recirculating system and thermostatically controlled

so that the temperature was held to within plus or minus two degrees of the desired temperature.

No attempt was made to measure or control the humidity in the oven, as Bewick (1) has found that in an oven of this type the relative humidity will be 20% plus or minus 2% at 125°F, over a wide range of relative humidity in the open air.

Each panel after proper preparation and drying, was masked with paper and tape so that one-half of the panel, approximately 5" x 16", was left exposed. This exposed area was coated with a paste consisting of 7 grams of FFF grade pumice stone and 7.5 cc. of paraffine oil and rubbed one hundred double strokes with a clean cloth pad. The panel was then wiped clean, coated with a paste consisting of 4 grams of powdered rottenstone and 7.5 cc. of paraffine oil, and rubbed one hundred double strokes with another clean cloth pad. The panel was then given a final polish with a pad of dry cheesecloth.

METHODS FOR TESTING PANELS

SET-TO-TOUCH TIME - Glass panels were prepared as previously described. The varnish film was rubbed lightly with the finger at five-minute intervals until no varnish adhered to the skin. The total time from the application of the varnish to the successful completion of the test was considered the set-to-touch time.

TACK-FREE TIME - The same glass panels were used for this determination. The varnish film was touched lightly with the finger tip at five minute intervals until it ceased to be tacky or sticky to the touch. The total time from the application of the varnish to the successful completion of the test was considered the tack-free time.

ADHESION OF VARNISH TO WOOD PANELS - The adhesion of the varnish film to the underlying sealer was determined by its resistance to the removal of the film by the thumb nail.

PRINT TEST - This test was made by placing a square of double thickness cheesecloth on the freshly rubbed panel and covering this square with a flat bottomed metal pan. This pan was weighted to give a definite weight per square inch of the area of the bottom of the pan. The weights used were $\frac{1}{2}$, 1, $1\frac{1}{2}$, and 2 pounds per square inch.

These weights were allowed to remain for twenty-four hours; then they were removed and the degree of printing noted.

ALCOHOL TEST - One cc. of 100 proof bourbon whiskey was placed on the rubbed portion of the panel and covered with an inverted flat bottomed metal pan identical with the pans used in the print test described above. These pans were allowed to remain on the panel for twenty-four hours, then removed, the panel wiped dry, and the effect of the whiskey on the varnish film recorded.

WATER TEST - This test was made in the same manner as the alcohol test, except that one cc. of tap water was substituted for the whiskey.

AGING TEST - The panels were exposed to the atmosphere at room temperature for a period of three months, then scratched with a spatula to determine the relative flexibility and adhesion of the various films.

EXPERIMENTAL

EXPERIMENTAL DATA

In order to determine the relative merits of varnishes containing ethyl cellulose which were made by incorporating the desired amount of ethyl cellulose during the cooking procedure, and those made by blending a varnish of high ethyl cellulose content with one containing no ethyl cellulose, a preliminary comparison of the following varnishes was made:

V-5-6 (Cook) vs. V-5-6 (Blend)

V-15-6 (Cook) vs. V-15-6 (Blend)

V-25-6 (Cook) vs. V-25-6 (Blend)

This comparison included the determination of the set-to-touch time, the tack-free time, the dry, adhesion and hardness of the film after 24 hours drying at room temperature, and print, alcohol and water tests.

RESULTS AND CONCLUSIONS

DRY AND ADHESION - The set-to-touch and tack-free times of the six varnishes are shown in Table II. These data are shown graphically in Figure 1. In each case the blended varnishes had slightly better through-dry, adhesion, and hardness on wood panels than the straight cooked varnishes.

PRINT TEST - The results of the print test are shown in Table III.

ALCOHOL AND WATER TESTS - The results of the alcohol and water tests are shown in Table IV.

The results of this preliminary investigation showed a slight but definite difference in properties between the blended varnishes and the straight cooked varnishes.

The blended varnishes and the straight cooked varnishes had similar set-to-touch times, but the tack-free times of the blends were less than the tack-free times of the corresponding cooks by approximately 20-22%.

TABLE II
SET-TO-TOUCH AND TACK-FREE TIME
PRELIMINARY EXPERIMENTAL

VARNISH	SET-TO-TOUCH TIME - MINUTES	TACK-FREE TIME - MINUTES
V-5-6 (Blend)	50	200
V-5-6 (Cook)	50	250
V-15-6 (Blend)	35	110
V-15-6 (Cook)	35	140
V-25-6 (Blend)	20	70
V-25-6 (Cook)	20	90

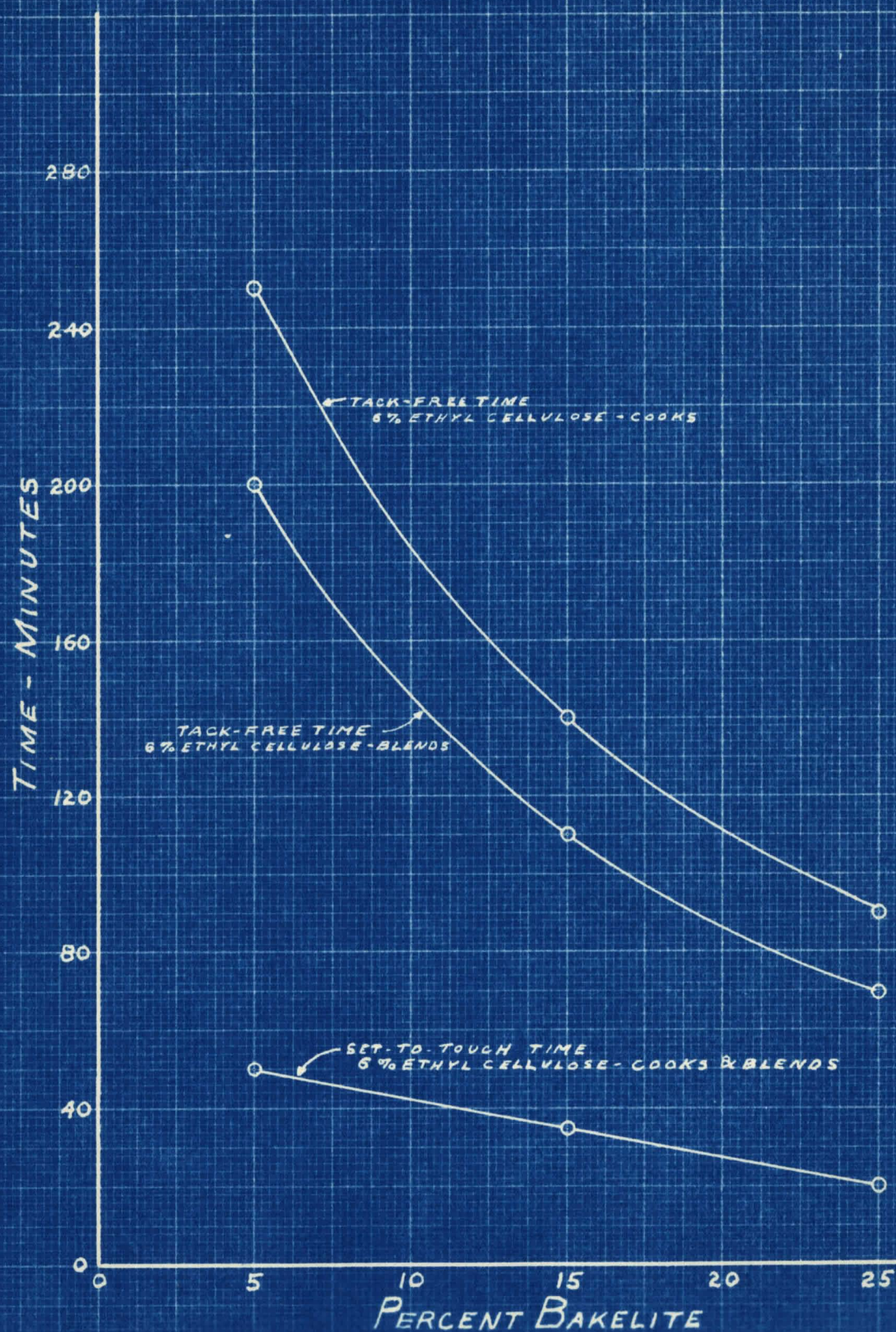


FIGURE 1 - SET-TO-TOUCH AND TACK-FREE TIMES
PRELIMINARY EXPERIMENTAL

TABLE III

PRINT TEST - 1 LB. PER SQ. IN.

PRELIMINARY EXPERIMENTAL

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-6 (Blend)	C	C
V-5-6 (Cook)	B	B
V-15-6 (Blend)	S	C
V-15-6 (Cook)	C	B
V-25-6 (Blend)	S	C
V-25-6 (Cook)	C	B

TABLE IV

ALCOHOL AND WATER TESTS
PRELIMINARY EXPERIMENTAL

VARNISH	ALCOHOL TEST	WATER TEST
V-5-6 (Blend)	VSS	H
V-5-6 (Cook)	SS	H
V-15-6 (Blend)	VSS	H
V-15-6 (Cook)	SS	H
V-25-6 (Blend)	H	H
V-25-6 (Cook)	VSS	H

Although there was no noticeable difference in water resistance between the varnishes prepared by the two methods, the print test and alcohol test indicated that the blends were slightly superior to the corresponding cooked varnishes.

These results were conclusive and indicated that the remainder of this problem should be the investigation of : 1 - Those varnishes containing a maximum of ethyl cellulose; 2 - Those containing no ethyl cellulose; and 3 - Varnishes of intermediate ethyl cellulose content prepared by blending 1 and 2.

EXPERIMENTAL DATA

TABLE V
SET-TO-TOUCH AND TACK-FREE TIMES
AVERAGE OF THREE DETERMINATIONS

VARNISH	SET-TO-TOUCH TIME - MINUTES	TACK-FREE TIME - MINUTES
V-5-0 *	42.5 #	180.0 #
V-5-4	35.0	160.0
V-5-6	33.3	146.6
V-5-8	31.6	130.0
V-5-10	26.6	116.6
V-5-12	23.3	96.6
V-15-0	27.5 #	75.0 #
V-15-4	25.0	75.0
V-15-6	25.0	75.0
V-15-8	25.0	75.0
V-15-10	25.0	70.0
V-15-12	22.5 #	60.0 #
V-25-0	20.0	50.0
V-25-4	20.0	50.0
V-25-6	20.0	50.0
V-25-8	20.0	50.0
V-25-10	20.0	50.0
V-25-12	20.0	45.0

Average of two determinations.

* V-5-0 is the designation for a varnish (V) with 5% of resin as Bakelite BR 3360 and 95% as ester gum (5) and 0% ethyl cellulose.

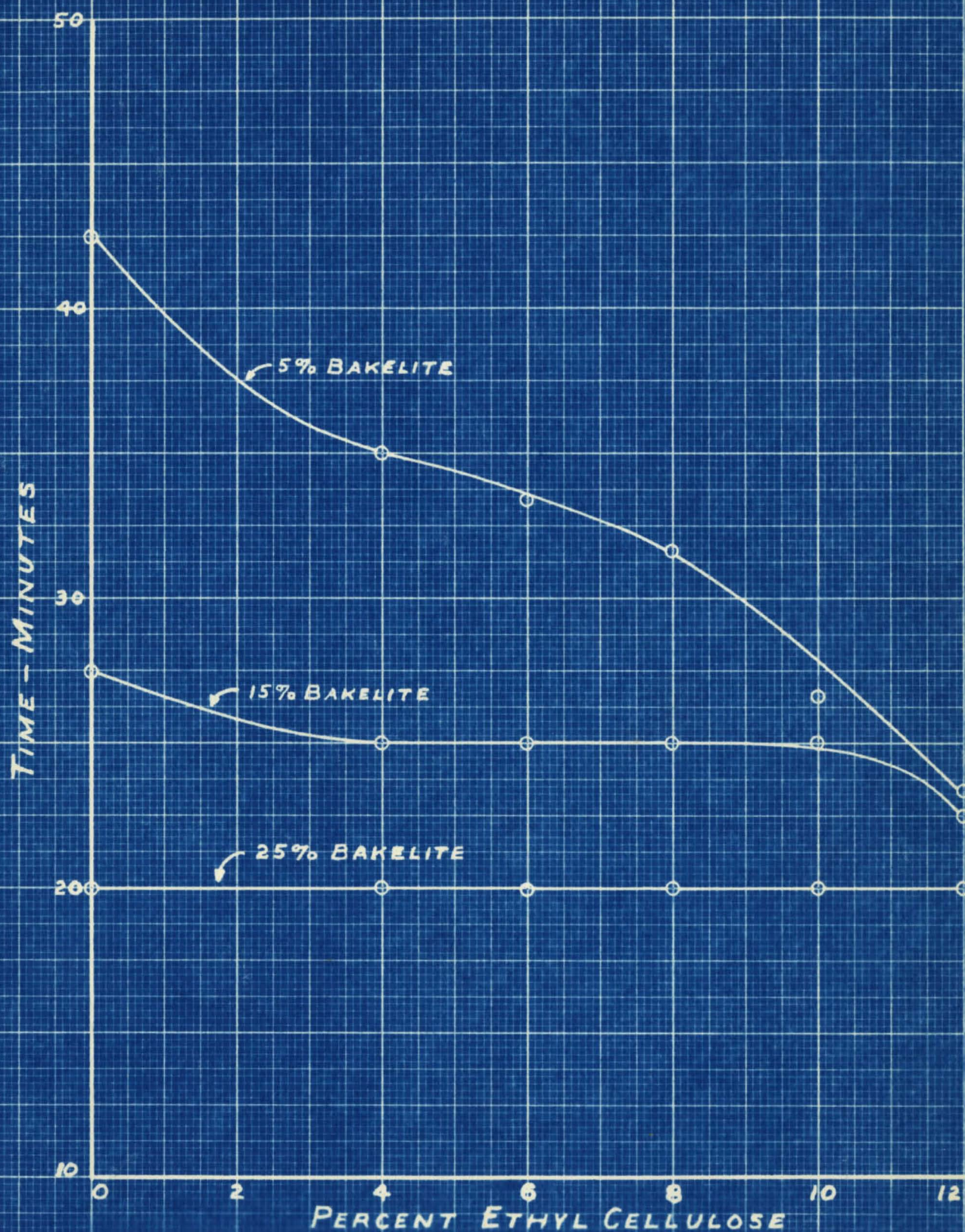


FIGURE 2 - SET-TO-TOUCH TIMES

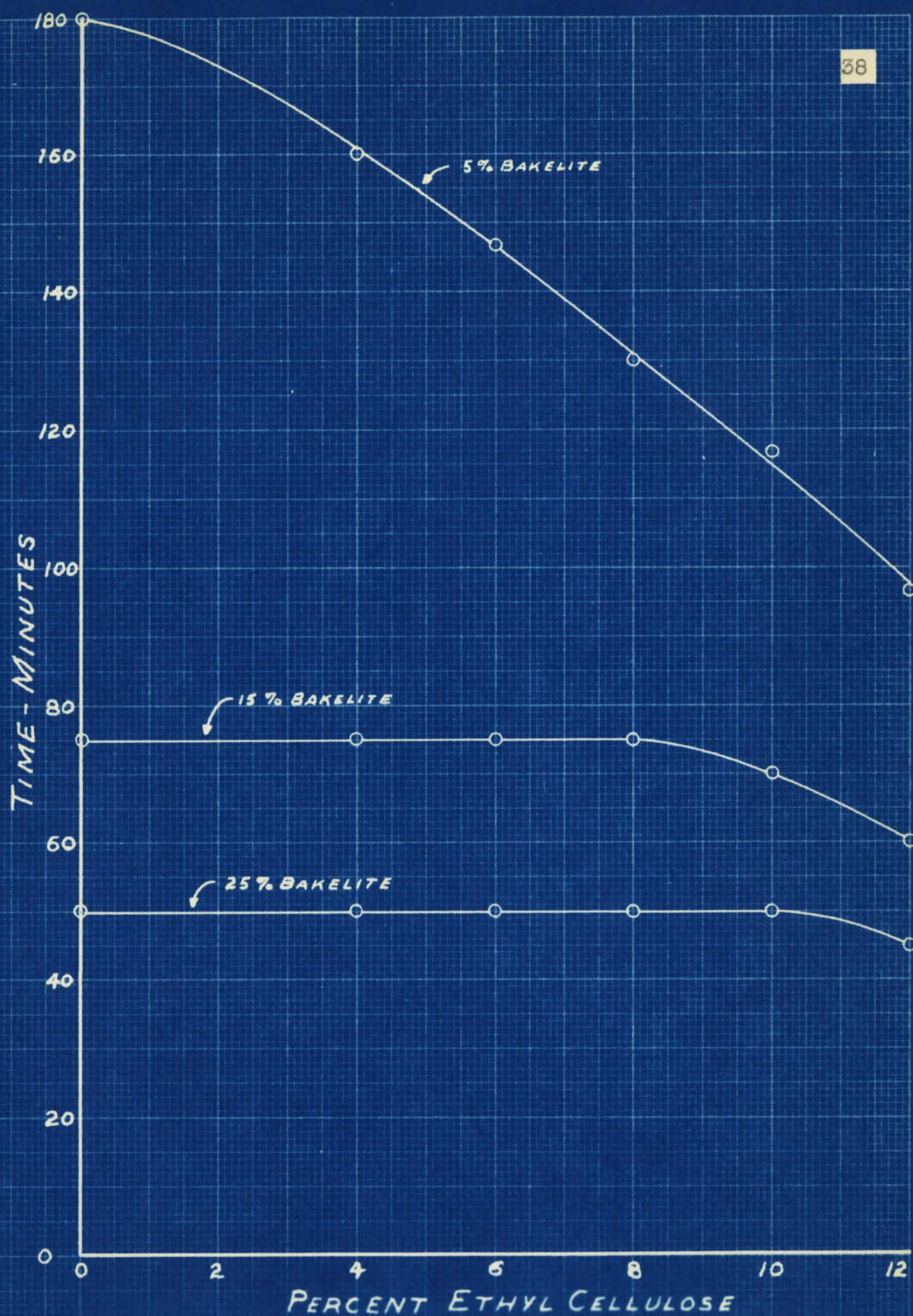


FIGURE 3 - TACK-FREE TIMES

TABLE VI

ADHESION

PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

VARNISH	ADHESION
V-5-0	F
V-5-4	G
V-5-6	G
V-5-8	G
V-5-10	G
V-5-12	G
V-15-0	P
V-15-4	G
V-15-6	G
V-15-8	G
V-15-10	G
V-15-12	G
V-25-0	VP
V-25-4	P
V-25-6	G
V-25-8	G
V-25-10	G
V-25-12	G

TABLE VII
ADHESION
PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F

VARNISH	ADHESION
V-5-0	P
V-5-4	G
V-5-6	G
V-5-8	G
V-5-10	G
V-5-12	G
V-15-0	P
V-15-4	G
V-15-6	G
V-15-8	G
V-15-10	G
V-15-12	G
V-25-0	VP
V-25-4	P
V-25-6	G
V-25-8	G
V-25-10	G
V-25-12	G

TABLE VIII

PRINT TEST - $\frac{1}{2}$ LB. PER SQ. IN.

PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	B	B
V-5-4	C	E
V-5-6	C	C
V-5-8	S	C
V-5-10	S	C
V-5-12	VS	S
V-15-0	C	E
V-15-4	S	C
V-15-6	S	C
V-15-8	VS	S
V-15-10	N	VS
V-15-12	N	VS
V-25-0	S	C
V-25-4	VS	S
V-25-6	VS	S
V-25-8	VS	S
V-25-10	N	VS
V-25-12	N	VS

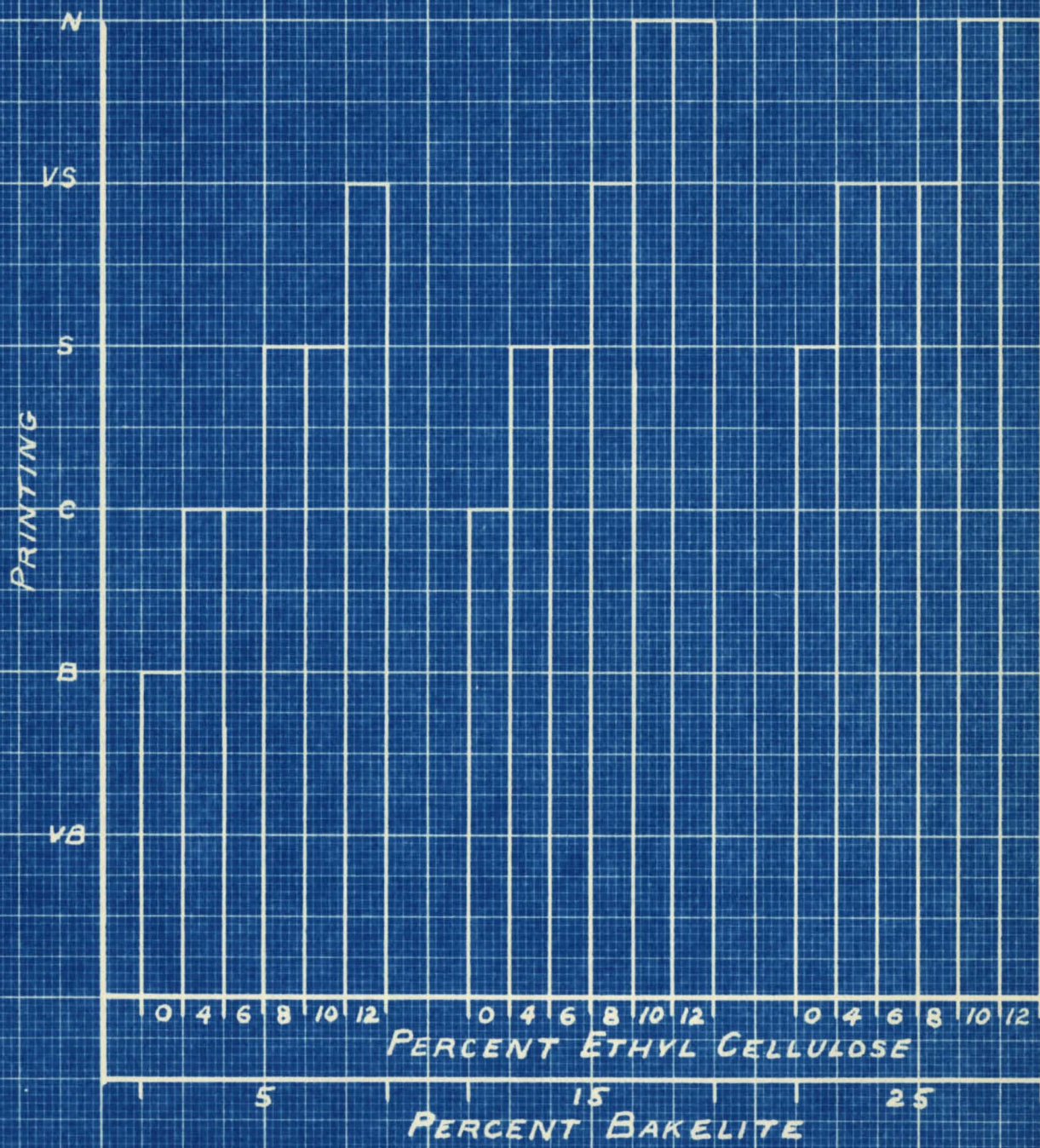


FIGURE 4 - PRINT TEST - $\frac{1}{2}$ LB. PER SQ. IN. ON RUBBED PORTION OF PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

EUGENE DIETZGEN CO.
PRINTED IN U.S.A.

NO. 340-20 DIETZGEN GRAPH PAPER
20 X 20 PER INCH

TABLE IX
PRINT TEST - 1 LB. PER SQ. IN.
PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	B	B
V-5-4	B	B
V-5-6	B	B
V-5-8	C	B
V-5-10	C	C
V-5-12	S	C
V-15-0	B	B
V-15-4	C	B
V-15-6	C	B
V-15-8	S	C
V-15-10	VS	S
V-15-12	N	VS
V-25-0	C	B
V-25-4	S	C
V-25-6	S	C
V-25-8	VS	S
V-25-10	N	VS
V-25-12	N	VS

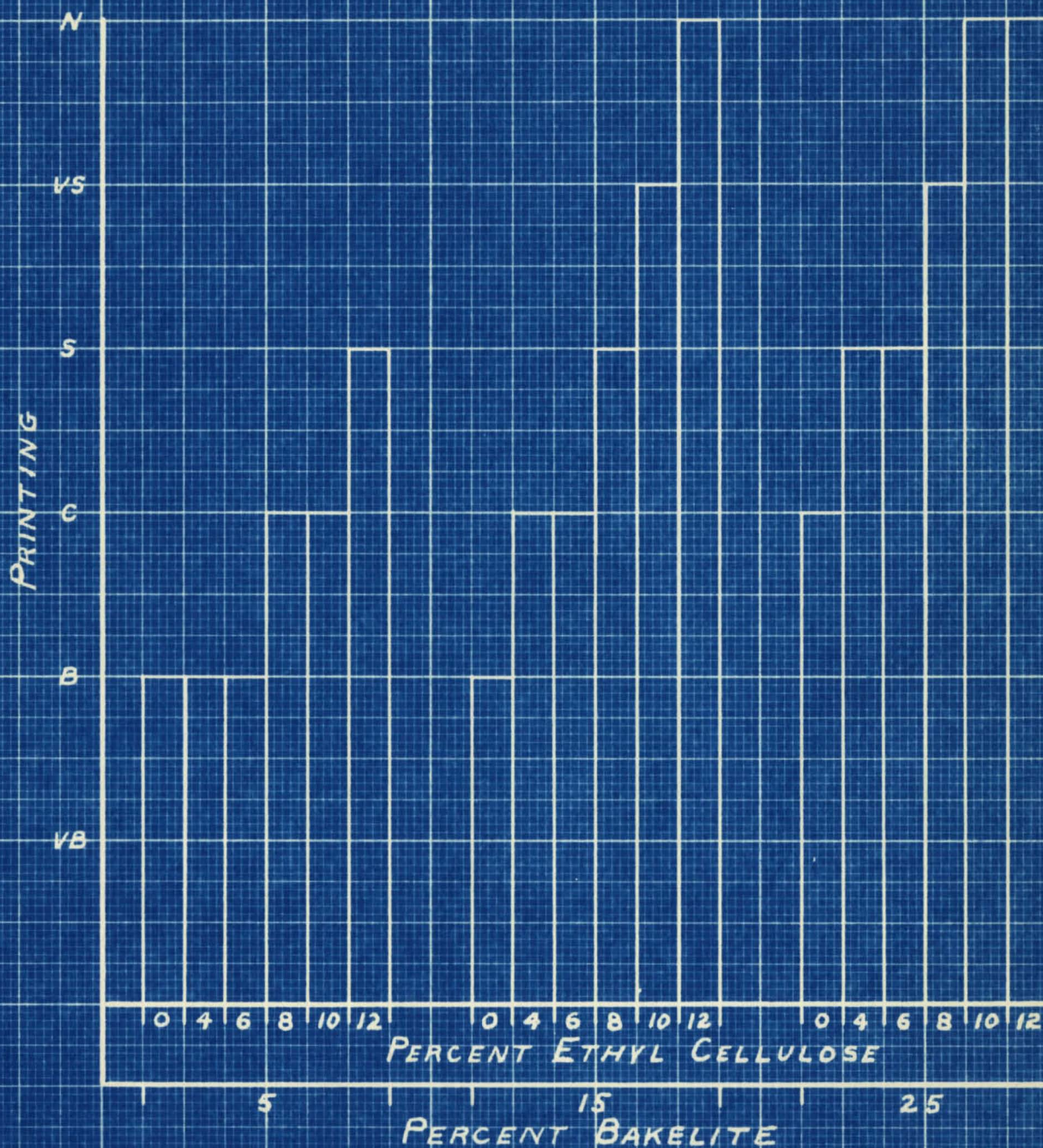


TABLE X

PRINT TEST - $1\frac{1}{2}$ LB. PER SQ. IN.

PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	B	B
V-5-4	B	B
V-5-6	B	B
V-5-8	B	B
V-5-10	C	B
V-5-12	S	C
V-15-0	B	B
V-15-4	C	B
V-15-6	C	B
V-15-8	S	C
V-15-10	VS	S
V-15-12	VS	S
V-25-0	C	B
V-25-4	S	C
V-25-6	S	C
V-25-8	S	C
V-25-10	VS	S
V-25-12	N	VS

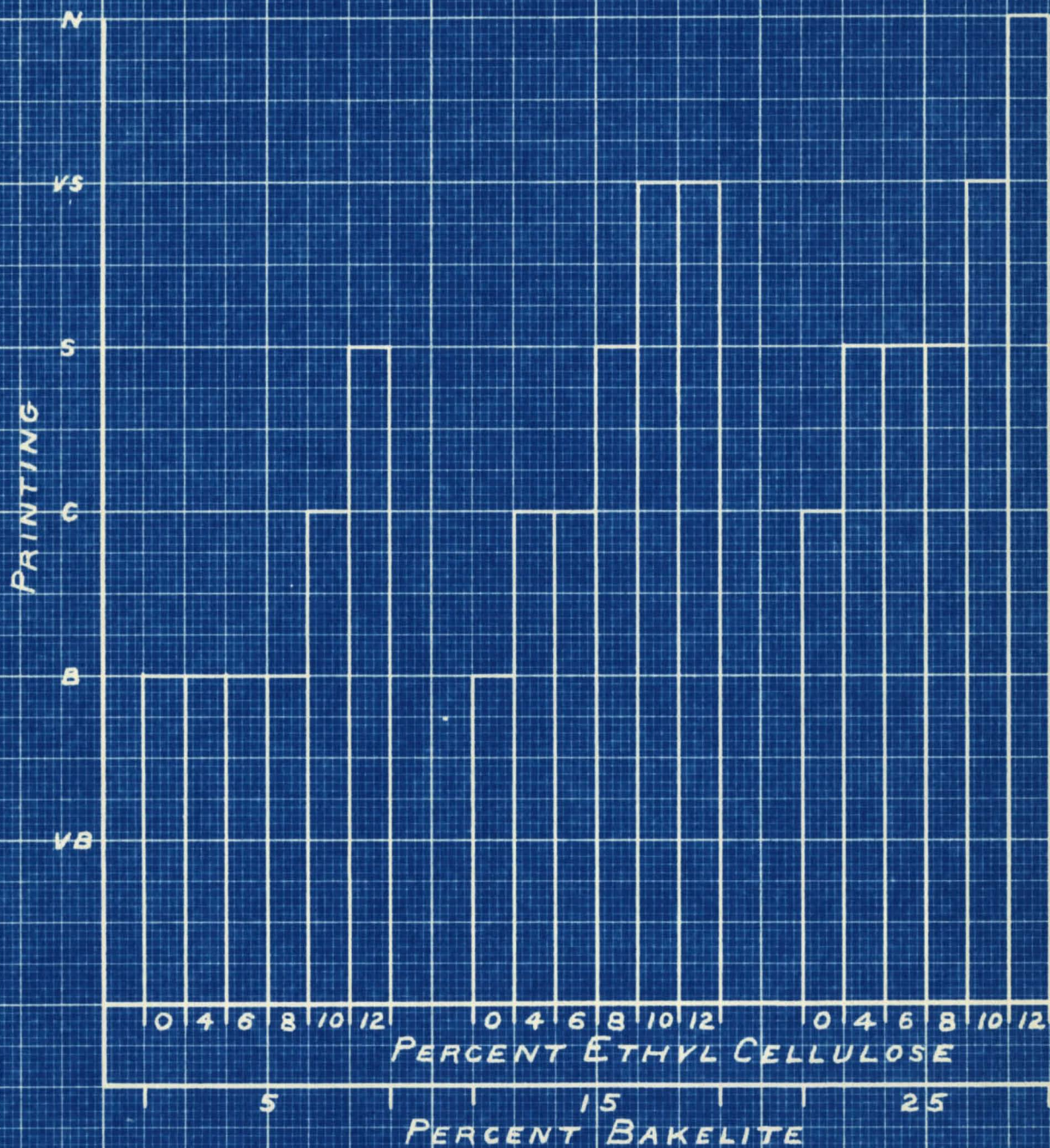


FIGURE 6 - PRINT TEST - $1\frac{1}{2}$ LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

TABLE XI

PRINT TEST - 1 LB. PER SQ. IN.
 PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F

VAHNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	C	B
V-5-4	C	B
V-5-6	S	C
V-5-8	VS	S
V-5-10	VS	S
V-5-12	VS	S
V-15-0	C	B
V-15-4	C	B
V-15-6	S	C
V-15-8	VS	S
V-15-10	VS	S
V-15-12	VS	S
V-25-0	S	B
V-25-4	S	B
V-25-6	VS	C
V-25-8	N	S
V-25-10	N	S
V-25-12	N	S

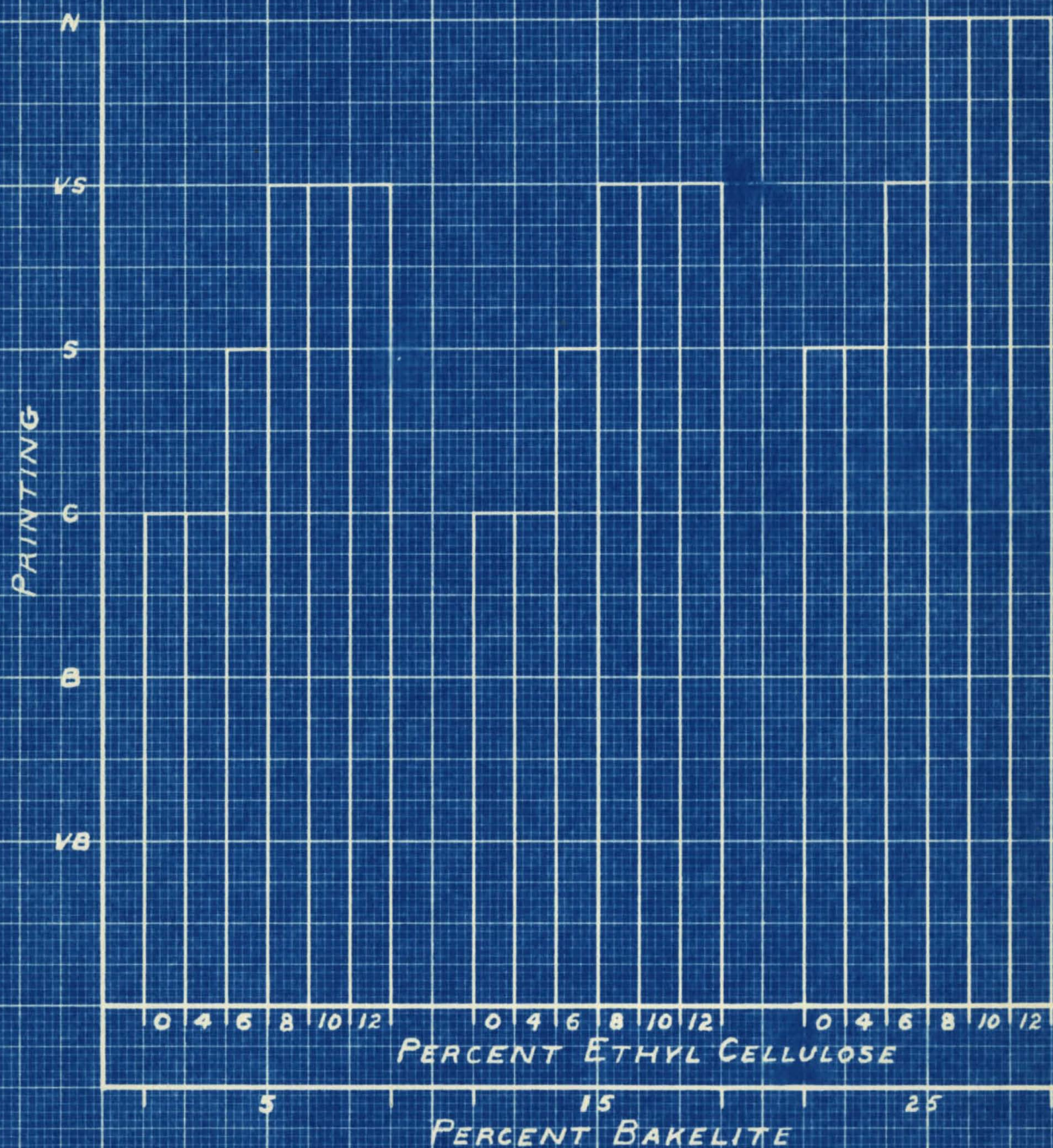


FIGURE 7 - PRINT TEST - 1 LB. PER SQ. IN. ON RUBBED
PORTION OF PANELS BAKED $1\frac{1}{2}$ HOURS
AT 150° F.

TABLE XII

PRINT TEST - $1\frac{1}{2}$ LBS. PER SQ. IN.PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	B	B
V-5-4	C	B
V-5-6	C	B
V-5-8	S	C
V-5-10	S	C
V-5-12	VS	S
V-15-0	B	B
V-15-4	C	B
V-15-6	C	B
V-15-8	S	C
V-15-10	S	C
V-15-12	VS	S
V-25-0	C	B
V-25-4	S	B
V-25-6	S	B
V-25-8	VS	C
V-25-10	VS	C
V-25-12	N	S

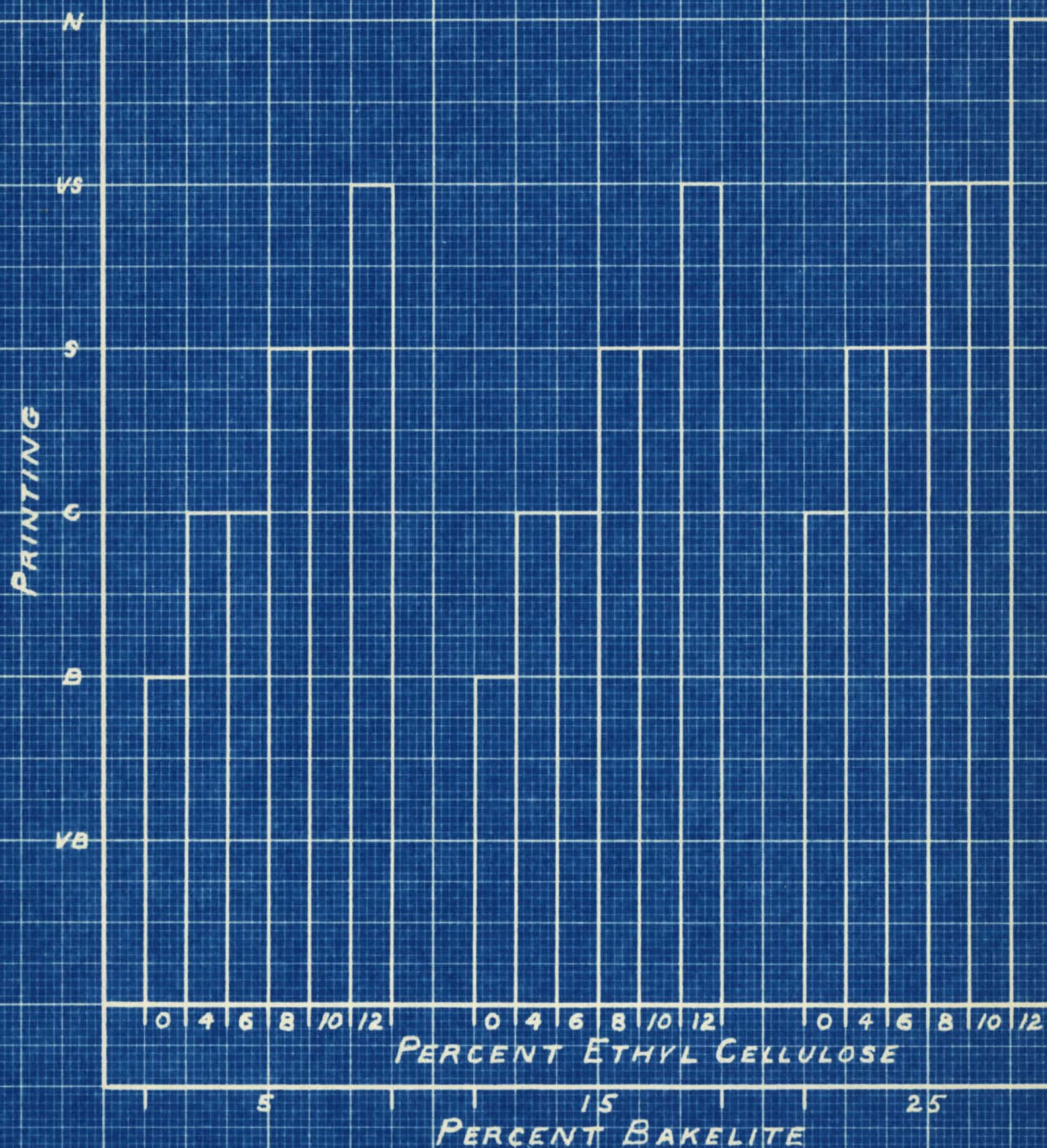


FIGURE 8 - PRINT TEST - $1\frac{1}{2}$ LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F.

TABLE XIII

PRINT TEST - 2 LBS. PER SQ. IN.

PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	B	B
V-5-4	B	B
V-5-6	C	B
V-5-8	C	B
V-5-10	S	C
V-5-12	S	C
V-15-0	B	B
V-15-4	B	B
V-15-6	C	B
V-15-8	C	B
V-15-10	S	C
V-15-12	S	C
V-25-0	C	B
V-25-4	C	B
V-25-6	S	B
V-25-8	S	B
V-25-10	VS	C
V-25-12	VS	C

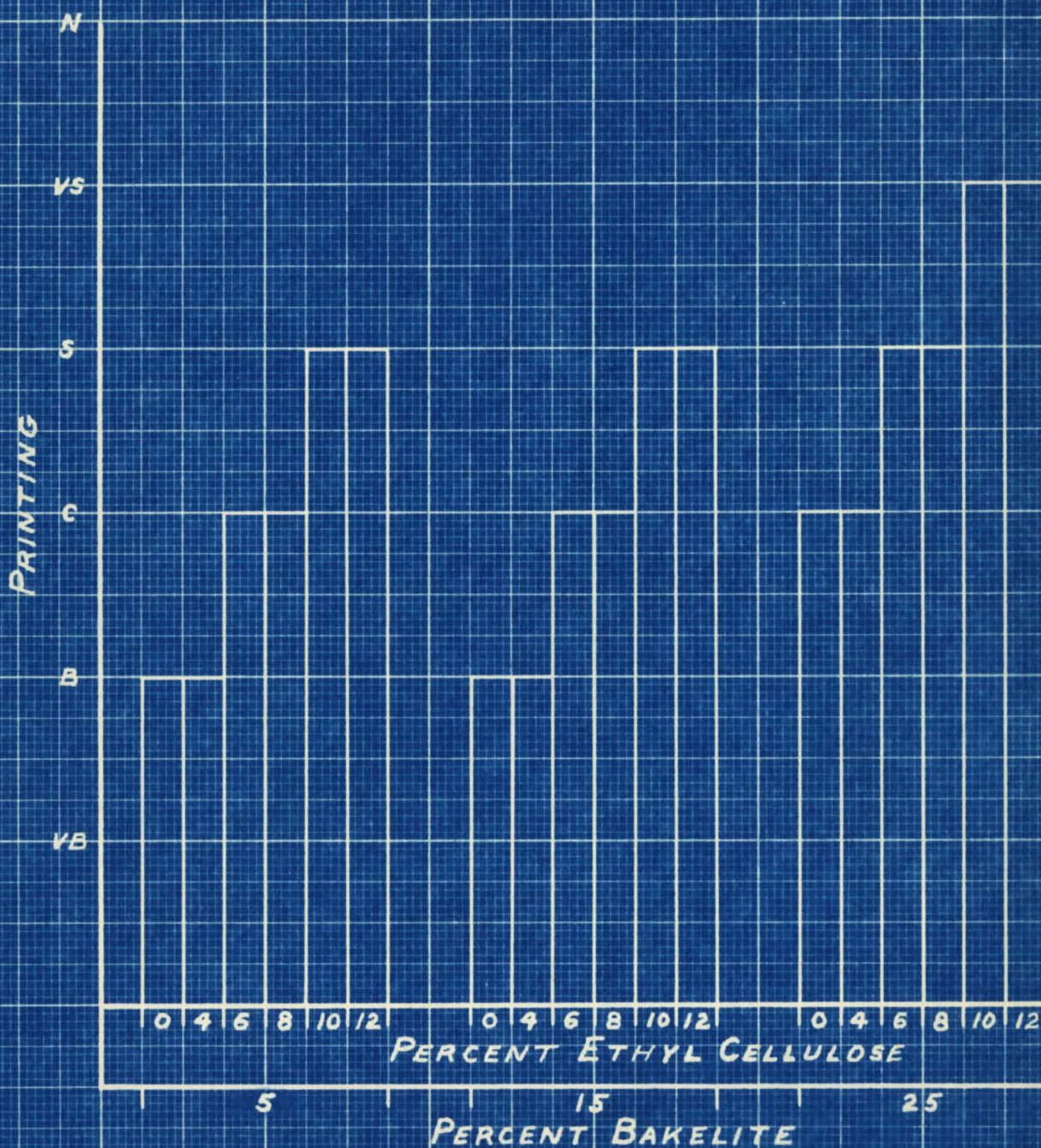


FIGURE 9 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F.

TABLE XIV
PRINT TEST - 2 LBS. PER SQ. IN.
PANELS FORCE-DRIED 2 HOURS AT 120° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	VB	VB
V-5-4	VB	VB
V-5-6	VB	VB
V-5-8	VB	VB
V-5-10	VB	VB
V-5-12	VB	VB
V-15-0	VB	VB
V-15-4	VB	VB
V-15-6	VB	VB
V-15-8	VB	VB
V-15-10	B	VB
V-15-12	B	VB
V-25-0	VB	VB
V-25-4	VB	VB
V-25-6	VB	VB
V-25-8	B	VB
V-25-10	C	B
V-25-12	C	B

FIGURE 10 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED
PORTION OF PANELS FORCE-DRIED 2 HOURS
AT 120° F.

TABLE XV
PRINT TEST - 2 LBS. PER SQ. IN.
PANELS FORCE-DRIED 3 HOURS AT 120° F

VARIATION	RUBBED PORTION	UNRUBBED PORTION
V-5-0	VB	VB
V-5-4	VB	VB
V-5-6	VB	VB
V-5-8	VB	VB
V-5-10	B	VB
V-5-12	B	VB
V-15-0	VB	VB
V-15-4	VB	VB
V-15-6	B	VB
V-15-8	B	VB
V-15-10	C	B
V-15-12	C	B
V-25-0	VB	VB
V-25-4	B	VB
V-25-6	B	VB
V-25-8	C	B
V-25-10	S	C
V-25-12	S	C

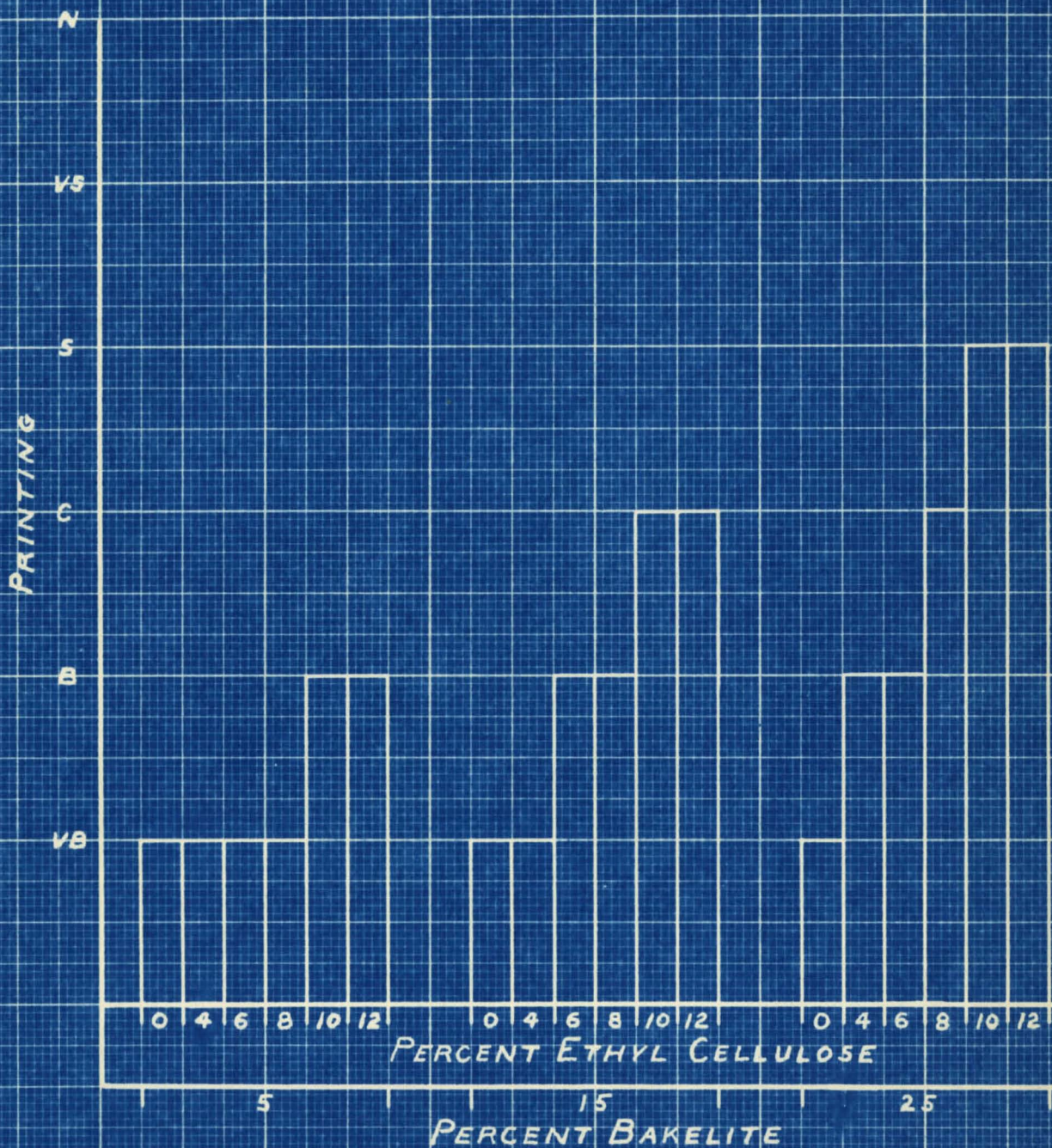


FIGURE 11 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS FORCE-DRIED 3 HOURS AT 120° F.

TABLE XVI

PRINT TEST - 2 LBS. RUB SQ. IN.

PANELS FORCE-DRIED 4 HOURS AT 120° F

VARVISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	VB	VB
V-5-4	B	VB
V-5-6	B	VB
V-5-8	B	VB
V-5-10	C	B
V-5-12	C	B
V-15-0	VB	VB
V-15-4	B	VB
V-15-6	B	VB
V-15-8	C	B
V-15-10	S	C
V-15-12	S	C
V-25-0	VB	VB
V-25-4	B	VB
V-25-6	C	B
V-25-8	S	C
V-25-10	VS	S
V-25-12	VS	S

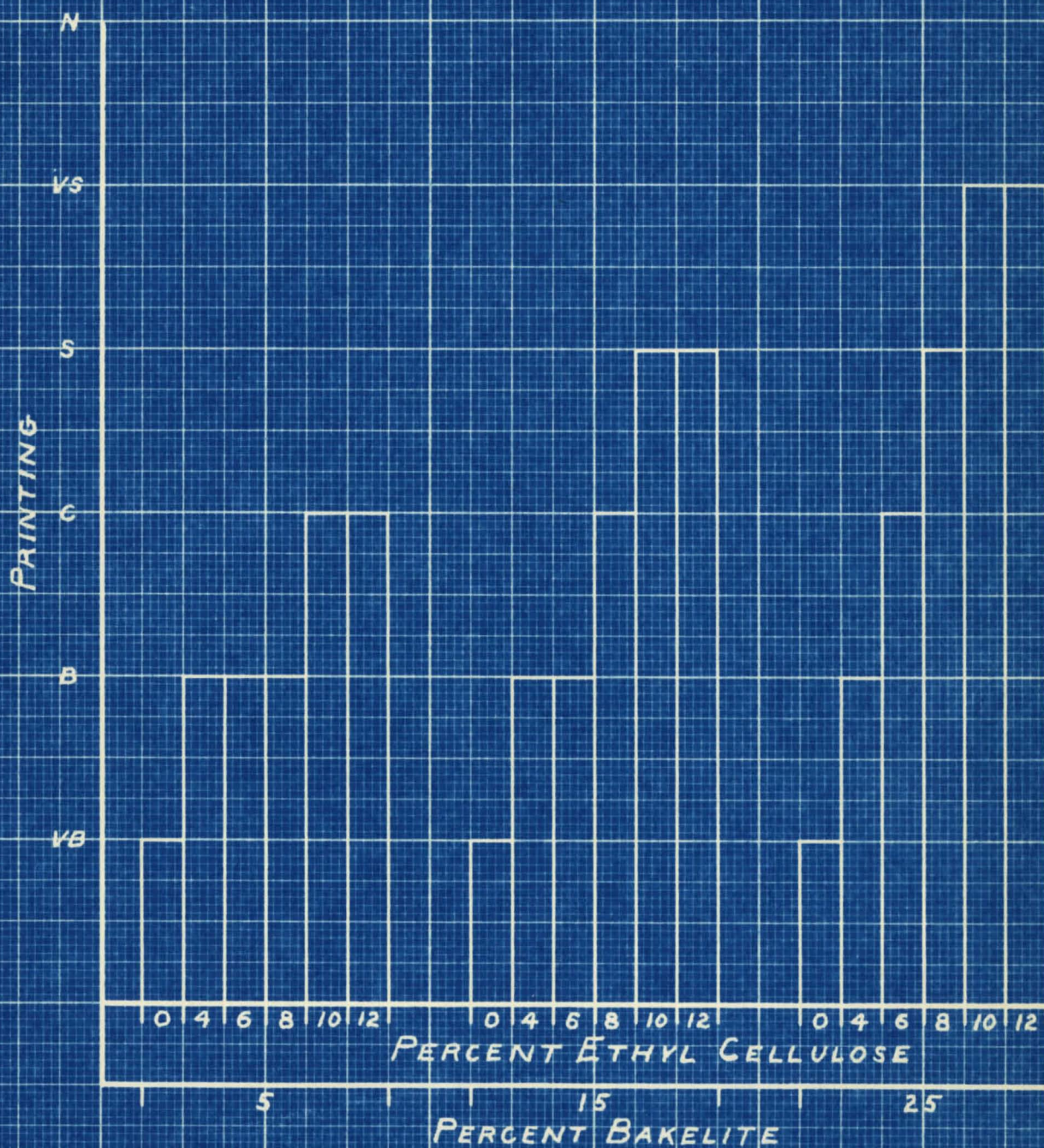


FIGURE 12 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED
PORTION OF PANELS FORCE-DRIED 4 HOURS
AT 120° F.

TABLE XVII

PRINT TEST - 2 LBS. PER SQ. IN.

PANELS FORCE-DRIED 5 HOURS AT 120° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	VB	VB
V-5-4	B	VB
V-5-6	C	B
V-5-8	C	B
V-5-10	S	C
V-5-12	S	C
V-15-0	VB	VB
V-15-4	B	VB
V-15-6	C	B
V-15-8	S	C
V-15-10	VS	S
V-15-12	VS	S
V-25-0	B	VB
V-25-4	C	B
V-25-6	S	C
V-25-8	VS	S
V-25-10	N	VS
V-25-12	N	VS

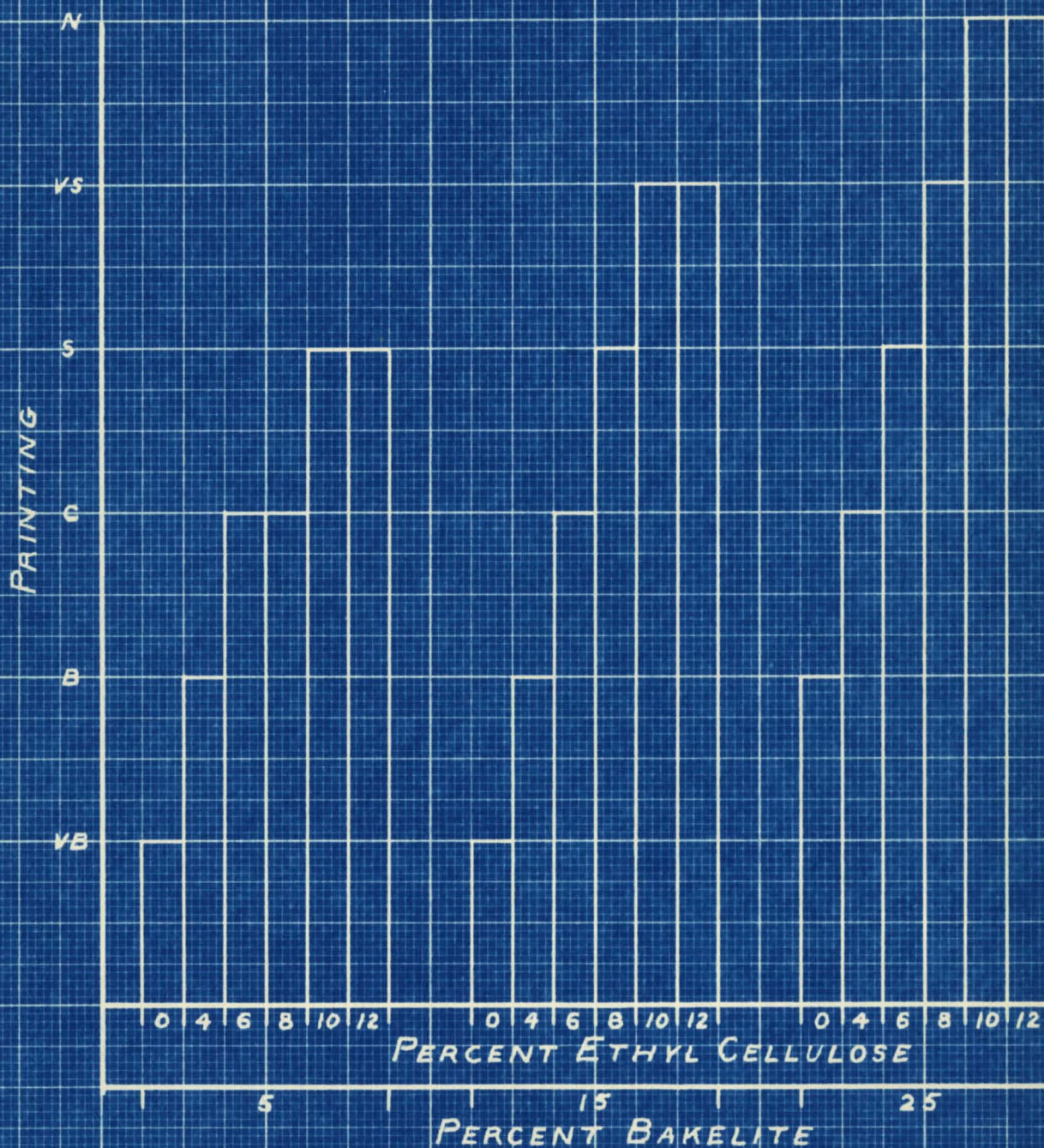


FIGURE 13 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS FORCE-DRIED 5 HOURS AT 120° F.

TABLE XVIII

PRINT TEST - 2 LBS. PER SQ. IN.

PANELS FORCE-DRIED 6 HOURS AT 120° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	VB	VB
V-5-4	C	B
V-5-6	C	B
V-5-8	S	C
V-5-10	VS	S
V-5-12	VS	S
V-15-0	B	VB
V-15-4	C	B
V-15-6	S	C
V-15-8	VS	S
V-15-10	VS	S
V-15-12	VS	S
V-25-0	C	B
V-25-4	S	C
V-25-6	VS	S
V-25-8	N	VS
V-25-10	N	VS
V-25-12	N	VS

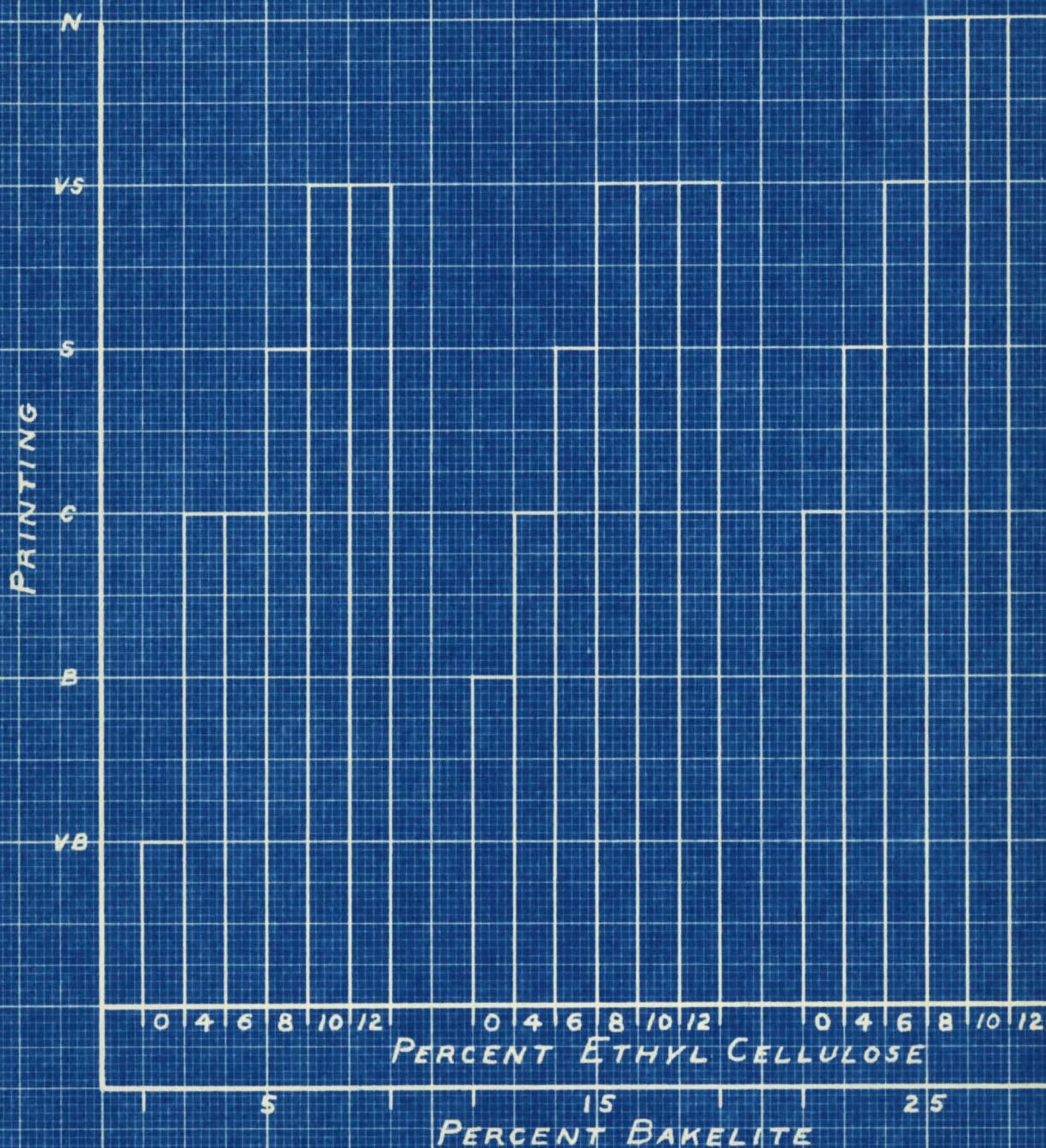


FIGURE 14 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS FORCE-DRIED 6 HOURS AT 120° F.

TABLE XIX
PRINT TEST - 2 LBS. PER SQ. IN.
PANELS FORCE-DRIED 7 HOURS AT 120° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	B	VB
V-5-4	C	B
V-5-6	S	C
V-5-8	VS	S
V-5-10	VS	S
V-5-12	VS	S
V-15-0	C	B
V-15-4	S	C
V-15-6	VS	S
V-15-8	VS	S
V-15-10	N	VS
V-15-12	N	VS
V-25-0	S	C
V-25-4	VS	S
V-25-6	N	VS
V-25-8	N	VS
V-25-10	N	N
V-25-12	N	N

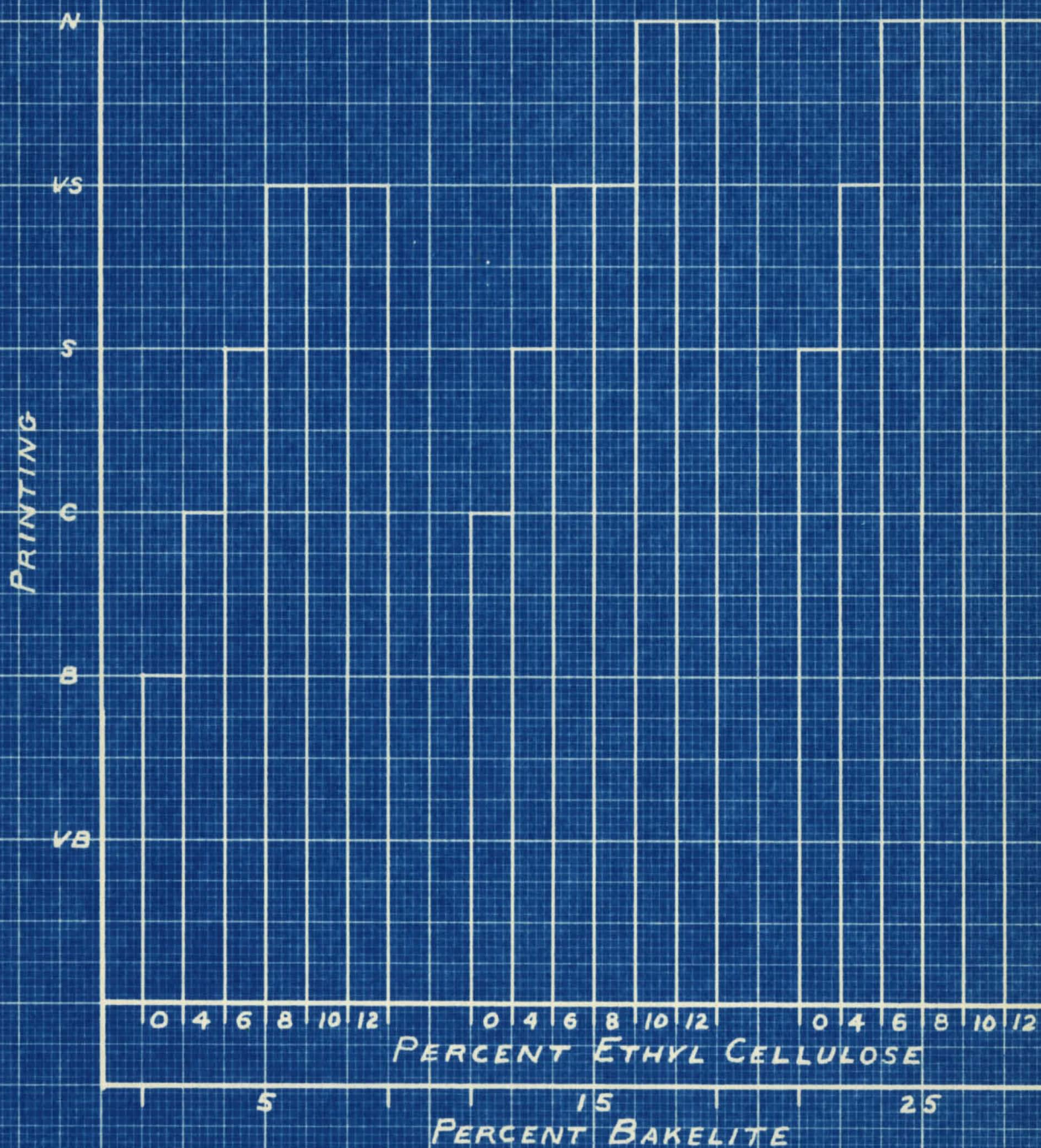


FIGURE 15 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS FORCE-DRIED 7 HOURS AT 120° F.

TABLE XX

PRINT TEST - 2 LBS. PER SQ. IN.

PANELS FORCE-DRIED 8 HOURS AT 120° F

VARNISH	RUBBED PORTION	UNRUBBED PORTION
V-5-0	C	B
V-5-4	S	C
V-5-6	S	C
V-5-8	VS	S
V-5-10	N	VS
V-5-12	N	VS
V-15-0	S	C
V-15-4	S	C
V-15-6	VS	S
V-15-8	N	VS
V-15-10	N	VS
V-15-12	N	VS
V-25-0	S	C
V-25-4	N	VS
V-25-6	N	VS
V-25-8	N	N
V-25-10	N	N
V-25-12	N	N

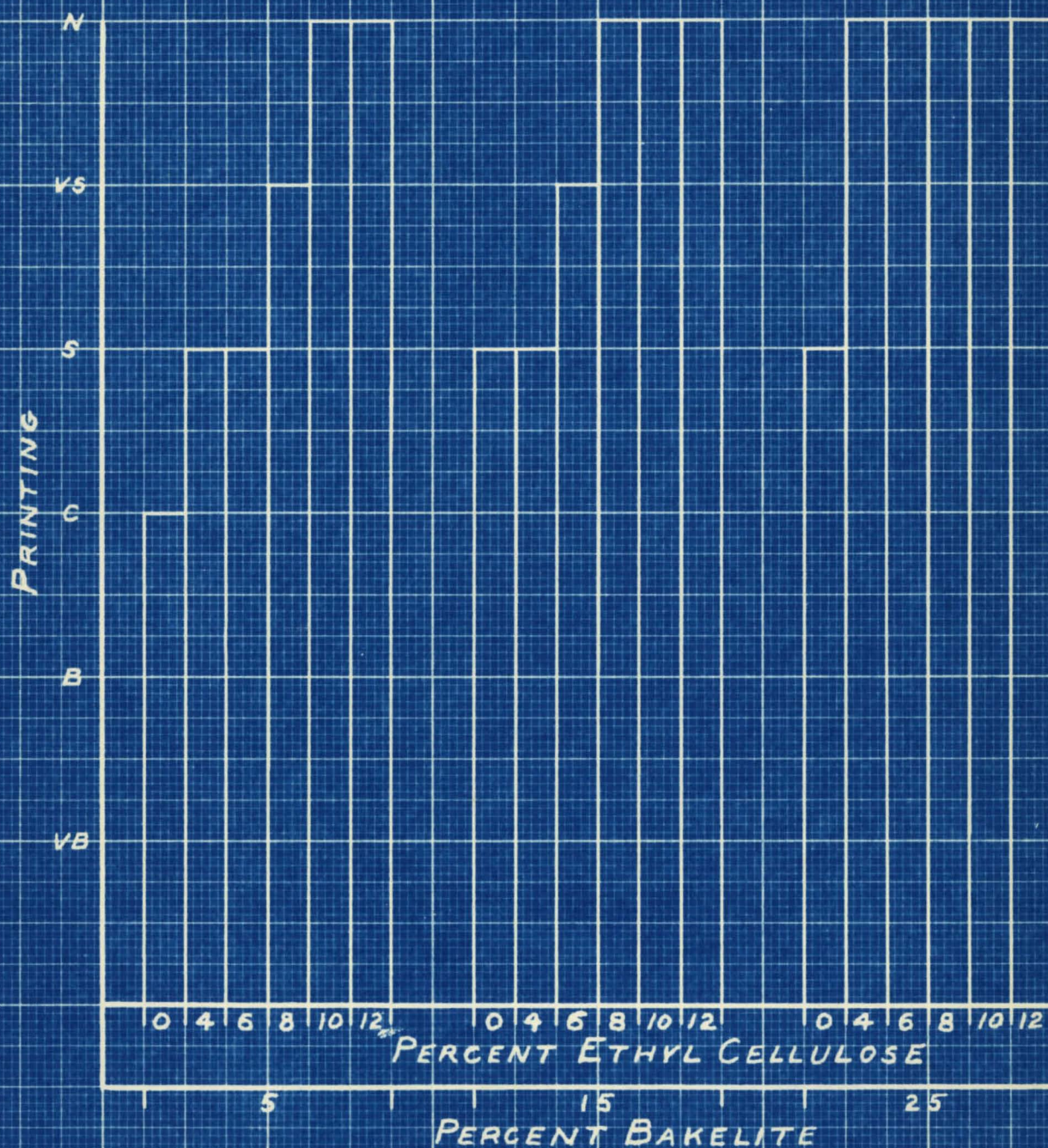


FIGURE 16 - PRINT TEST - 2 LBS. PER SQ. IN. ON RUBBED PORTION OF PANELS FORCE-DRIED 8 HOURS AT 120° F.

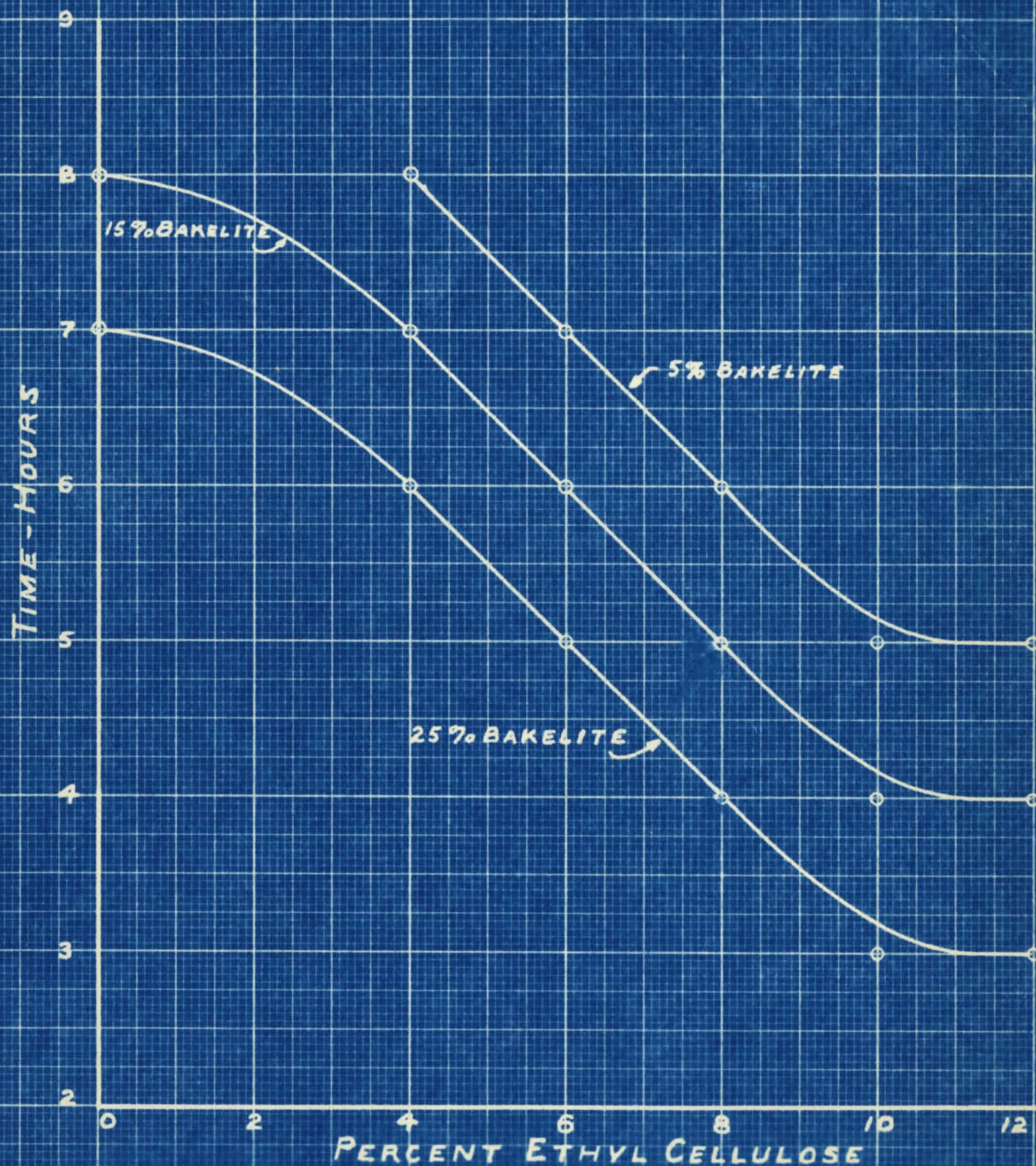


FIGURE 17 - FORCE-DRYING TIMES AT 120° F. TO GIVE A RATING OF "S" AT A PRINT WEIGHT OF 2 LBS. PER SQ. IN.

TABLE XXI

WATER TEST

PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

VARNISH	CONDITION OF FILM
V-5-0	S
V-5-4	SS
V-5-6	VSS
V-5-8	VSS
V-5-10	H
V-5-12	H
V-15-0	SS
V-15-4	VSS
V-15-6	H
V-15-8	H
V-15-10	H
V-15-12	H
V-25-0	SS
V-25-4	VSS
V-25-6	H
V-25-8	H
V-25-10	H
V-25-12	H

TABLE XXII
WATER TEST
PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F

VARNISH	CONDITION OF FILM
V-5-0	SS
V-5-4	H
V-5-6	H
V-5-8	H
V-5-10	H
V-5-12	H
V-15-0	VSS
V-15-4	H
V-15-6	H
V-15-8	H
V-15-10	H
V-15-12	H
V-25-0	VSS
V-25-4	H
V-25-6	H
V-25-8	H
V-25-10	H
V-25-12	H

TABLE XXIII

ALCOHOL TEST

PANELS DRIED 24 HOURS AT ROOM TEMPERATURE

VARNISH	CONDITION OF FILM
V-5-0	VS
V-5-4	S
V-5-6	S
V-5-8	VSS
V-5-10	VSS
V-5-12	VSS
V-15-0	SS
V-15-4	VSS
V-15-6	H
V-15-8	H
V-15-10	H
V-15-12	H
V-25-0	SS
V-25-4	VSS
V-25-6	H
V-25-8	H
V-25-10	H
V-25-12	H

TABLE XXIV
ALCOHOL TEST
PANELS BAKED $1\frac{1}{2}$ HOURS AT 150° F

VARNISH	CONDITION OF FILM
V-5-0	SS
V-5-4	VSS
V-5-6	H
V-5-8	H
V-5-10	H
V-5-12	H
V-15-0	SS
V-15-4	VSS
V-15-6	H
V-15-8	H
V-15-10	H
V-15-12	H
V-25-0	SS
V-25-4	VSS
V-25-6	H
V-25-8	H
V-25-10	H
V-25-12	H

TABLE XXV
ADHESION AND FLEXIBILITY OF
VARNISH FILMS AFTER AGING

PANELS DRIED AT ROOM TEMPERATURE FOR THREE MONTHS

VARNISH	ADHESION	FLEXIBILITY
V-5-0	F	VP
V-5-4	G	P
V-5-6	G	F
V-5-8	G	F
V-5-10	G	G
V-5-12	G	G
V-15-0	P	VP
V-15-4	G	P
V-15-6	G	F
V-15-8	G	F
V-15-10	G	G
V-15-12	G	G
V-25-0	VP	VP
V-25-4	F	P
V-25-6	G	F
V-25-8	G	F
V-25-10	G	G
V-25-12	G	G

RESULTS AND CONCLUSIONS

Ethyl cellulose, when properly incorporated in the varnish, increases the drying rate, adhesion, print, water, and alcohol resistances of ten-gallon-long phenolic resin - ester gum - china wood oil varnishes.

The set-to-touch and tack-free times of a varnish of low phenolic resin content decrease with increasing ethyl cellulose content. However, the improvement in the initial drying rate becomes less as the phenolic resin content increases. With varnishes in which the phenolic resin is 25% of the total resin, the set-to-touch time shows no change with increasing ethyl cellulose content, and the tack-free time decreases only when the ethyl cellulose content is 12% or greater.

The adhesion of the varnishes is improved by the incorporation of as little as 4 to 6% of ethyl cellulose. Varnishes of greater phenolic resin content require the greater percentage of ethyl cellulose to have satisfactory adhesion.

An increase in the ethyl cellulose content of a varnish containing a definite percentage of phenolic resin is usually accompanied by an increase in the print resistance when dried under stated conditions of time and temperature. Conversely, varnishes of equal phenolic resin content, but

varying in ethyl cellulose content, require less time at a definite temperature to give the same degree of print resistance as the ethyl cellulose content is increased.

The presence of ethyl cellulose in a varnish of this type causes a slight improvement in the water and alcohol resistance. As the phenolic resin content is increased, less ethyl cellulose is required to give excellent resistance.

The retention of flexibility and adhesion of varnish films after prolonged aging is markedly improved by the addition of ethyl cellulose to the varnish. The best retention is shown by those varnishes containing the highest percentages of ethyl cellulose.

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